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SPACE RADIATION EFFECTS ON TEFLON FILMS. (U)

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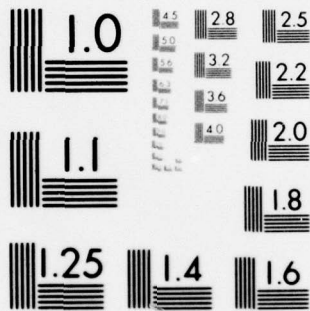
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Space Radiation Effects on Teflon Films

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Chemistry and Physics Laboratory
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Interim Report

28 September 1979

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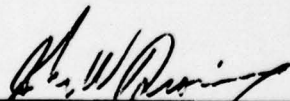
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This technical report has been reviewed and is approved for publication. Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

FOR THE COMMANDER



John W. Browning, Col, USAF
Director, International Satellite
Communications Program Office

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Degradation of macroscopic physical properties resulting from chemical changes in fluorocarbon polymers exposed to ionizing radiation is reviewed. The effects of temperature, ultraviolet/visible radiation, and dose rate are considered. Of particular interest are property changes that occur on exposure of these materials to simulated space radiation environments. Results from recent space simulation exposure tests are considered in light of these phenomena. General recommendations are made for the application			

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of existing data to spacecraft problems, as well as for the conduct of future space simulation exposure tests.

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PREFACE

The author gratefully acknowledges a number of helpful discussions with Robert J. Champetier of the Material Sciences Laboratory, particularly in reference to space simulation testing.

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I. INTRODUCTION

Utilization of silvered teflon films on spacecraft has led to considerable interest in property changes that occur upon exposure of these materials to the space environment. This interest has stimulated a number of laboratory studies on exposure of these polymers to simulated space environments. Results from these exposures have often produced misleading or contradictory results for a variety of reasons. Well known examples include the severe degradation of mechanical properties demonstrated in earlier studies and subsequently shown to be due to oxygen effects,^{1, 2} and bleaching of radiation induced coloration that occurred on exposure of vacuum irradiated samples to air.³

In this document chemical changes that occur on exposure of fluorocarbon polymers to various simulated elements of the space environment are reviewed. The effects of ionizing and ultraviolet/visible radiation as well as temperature and dose rate are considered. Although a great deal of literature exists on exposure of these materials to radiation in oxygen or air, these results are not generally of interest in this review and, except for a few areas, are not considered further. Unless otherwise noted, results discussed here are for polymers exposed to simulated environments in vacuum or inert gases, or for experiments in which exposures to air or oxygen were unimportant.

The FEP (fluorinated ethylene-propylene) polymers are of primary interest for space applications. However, most of the existing work has been carried out on TFE (tetrafluoroethylene) polymers. Where data exists, relevant work on FEP is reviewed. In other cases, TFE results are discussed and anticipated differences between FEP and TFE noted. In the following sections, chemical, charging, and structural changes observed upon exposure of fluorocarbon polymers to various environments are reviewed. The discussions consider events taking place on a molecular or micro scale and the impact the events have on macroscopic properties. The report then goes on to discuss the implication of these results with respect to recent

space simulation exposure tests, ^{4, 5} application of existing data to spacecraft problems, and the conduct of future experiments on space simulated testing of fluorocarbon polymer containing materials.

II. FLUOROCARBON POLYMER CHANGES ON EXPOSURE TO VARIOUS LABORATORY ENVIRONMENTS

A. Chemical Changes

1. Room Temperature Effects

In this section, chemical changes induced by exposure of these materials to selected environments are considered. These include the rupture of chemical bonds leading to the formation of free radicals (molecular fragments containing unpaired electrons) or unsaturated species (molecules possessing double bonds). Ionization and charge trapping phenomena are considered below (Section II. B.).

Chemical bond rupture can occur at either C-C bonds (chain scission) or C-F bonds leading to the formation of free radicals. Chain scission generally leads to degradation of mechanical properties such as tensile strength and elasticity ^{1,2}. Subsequent reactions between free radicals can lead to cross-linking in the polymers and increases in ultimate tensile strength and embrittlement. ^{1,2} Net changes in polymer properties will depend upon the relative importance of these processes. Additionally, the formation of unsaturated species can cause yellowing of the polymers.

Similar to the case of TFE (for excellent reviews, see Refs. 6 and 7; additional results, some of which are discussed in Refs. 6 and 7 may be found in Refs. 8 - 15), exposure of FEP to ionizing radiation at room temperature led to the formation of free radicals resulting from C-C and C-F bond ruptures ^{6,7,16,17} and having the structures $-\text{CF}_2^\bullet$ and $-\text{CF}_2\dot{\text{C}}\text{FCF}_2^-$. [Actually, the original work ^{16,17} indicated the former radical was $-\text{CF}_2\text{O}^\bullet$ not $-\text{CF}_2^\bullet$; however, work carried out at Aerospace ¹⁸ and elsewhere ¹⁹ demonstrated that this was based on a misinterpretation of the experimental results and the former radical was, indeed, $-\text{CF}_2^\bullet$]. In addition, a third radical was observed in FEP, whose concentration increased significantly with the fluorinated propylene content of the polymer. This species was

identified as $-\text{CF}_2\dot{\text{C}}(\text{CF}_3)\text{CF}_2-$, ^{16,17} as had earlier been suggested. ²⁰ In the presence of oxygen (air), these species readily react to form peroxy radicals. ^{6,7,21-35}

Examination of the experimental data indicated that, at low doses (< 10 Mrads), the total free radical yield for a given dose was an order of magnitude higher in FEP than TFE. ^{16,20,36} However, with increasing dose, the free radical yields in FEP leveled off while those in TFE continued to increase. ²⁶ In addition, radicals resulting from C-F rupture in both polymers were more than an order of magnitude more abundant than those resulting from C-C bond rupture. ^{6,7,16,17} This was somewhat surprising, in view of the higher strength of the C-F bond relative to that of the C-C bond (> 100 kcal/mole versus ~80 kcal/mole, respectively ³⁷).

This result was attributed to cage effects. ^{6,7,18,19} That is, C-C bond rupture does occur with a higher frequency than indicated by experimental results. However, this leads to the production of two high molecular weight free radicals that are unable to diffuse apart in the viscous polymers. Consequently, there is a high probability for geminate recombination of the two fragments. In contrast, the smaller fluorine atoms, produced by C-F bond ruptures, can readily diffuse through the polymers and combine with other diffusing fluorine atoms, thus stabilizing the higher molecular weight fragments.

Decreases in free radical yields from TFE and other polymers irradiated at high pressures (up to 10 kbar), where cage effects would be expected to increase, are consistent with this interpretation. ³⁸ So also are results from irradiations carried out in the presence of molecular chlorine. ³⁹ In the latter experiments, irradiation damage to TFE, as measured by deterioration of mechanical properties, was more severe when chlorine was present, relative to vacuum irradiation. In the presence of chlorine, free radical pairs formed by irradiation can react with the

molecular chlorine to form species that would preclude radical recombination, thus yielding greater net polymer degradation. Additionally, at low temperatures, one might expect the fragments from C-C bond scission to be frozen in and not recombine until the polymer is warmed somewhat to a point where molecular motions can facilitate recombination. Effects of this type have been observed for other polymers irradiated at liquid nitrogen temperatures.⁴⁰

The radicals described above appear to be generally stable to ultraviolet light exposures, in contrast to their oxygenated counterparts.^{6, 7, 18, 30} One exception to this is the low temperature reversible photoisomerization of the $-\text{CF}_2\dot{\text{C}}(\text{CF}_3)\text{CF}_2-$ radical in FEP to form $-\text{CF}_2\text{CF}(\dot{\text{C}}\text{F}_2)\text{CF}_2-$.^{7, 17} This isomerization would probably have little impact on macroscopic properties of the polymer.

A transformation that could impact macroscopic physical properties is that of $-\text{CF}_2\bullet$ radicals into $-\text{CF}_2\dot{\text{C}}\text{FCF}_2-$ upon exposure to 1.7 Mev electrons as suggested by Hedvig.⁴¹ [Work carried out at Aerospace⁴² and elsewhere⁴³ has demonstrated that oxygenated free radicals are extremely radiation sensitive.] However, the experimental results presented in Ref. 41 appear to be inconsistent with this interpretation. Although the $-\text{CF}_2\dot{\text{C}}\text{FCF}_2-$ concentration did increase during the electron exposure, so also did the $-\text{CF}_2\bullet$ and total radical yields. The complexity of the experiment and insufficient detail given do not permit an alternative explanation to be put forth.

Several studies have indicated the formation of unsaturated species in TFE polymers on exposure to ionizing radiation as inferred by infrared and ESR (electron spin resonance) spectroscopic investigations⁴⁴⁻⁴⁷ (work in the latter reference was carried out at sub-ambient temperatures); although an additional study indicated negative results.⁴⁸ The ESR studies^{46, 47} suggest the formation of fluorinated allylic free radicals of the type $-\text{CF}_2\text{CF}=\text{C}\dot{\text{C}}\text{FCF}_2-$. The positive infrared results could be due to these or other unsaturated polymer fragments. They could also be due to

occluded gases since several studies have indicated the formation of low molecular weight unsaturated fluorocarbons in both TFE and FEP upon irradiation.^{20,49} The latter results, along with the similarity in structures between FEP and TFE, suggest that unsaturated species could also be produced in irradiated FEP polymers.

Several studies have examined the effects of ultraviolet radiation (185 to 369 nm) and fluorocarbon polymers.⁵⁰⁻⁵⁴ Decreases in tensile strength and ultimate elongation were observed for TFE polymers exposed, at temperatures of -70 to 600°F, to ultraviolet radiation in vacuum or nitrogen, suggesting the occurrence of chain scission. Similar exposures of FEP films⁵⁴ yielded property changes suggestive of chain scission processes at ambient or slightly higher temperatures, and crosslinking above ~175°F.

2. Low Temperature Effects

Only one study has been carried out on FEP irradiated at low temperature (liquid nitrogen temperature¹⁷). In that study, the authors concluded that $-\text{CF}_2\dot{\text{C}}(\text{CF}_3)\text{CF}_2-$ was the primary radical formed. They also suggested the conversion of this radical into $-\text{CF}_2\text{CF}(\dot{\text{C}}\text{F}_2)\text{CF}_2-$ upon exposure to UV light, as described earlier.^{7,17} No information on radical yields was given.

Three different studies were carried out on irradiated TFE at liquid helium and/or liquid nitrogen temperatures.^{19,47,55} The results and interpretations of the results from these experiments were quite different. The observed ESR spectra were attributed predominantly to trapped charges, allylic type free radicals,⁴⁷ and $-\text{CF}_2\bullet$ radicals⁵⁵ in addition to species observed at room temperature. In the former study¹⁹ ultraviolet/visible photoinduced changes were observed at liquid nitrogen temperature. Discrepancies in the limited results presented precludes a more definitive identification. However, for purposes of the present exercise, most of the added species present at low temperature disappear on warming to -110°F and the remaining radicals are similar to those observed at room temperature.^{19,56}

3. High Temperature Effects

Examination of the literature indicates that free radicals are stable in both irradiated TFE^{55, 56} and FEP¹⁶ polymers from room temperature up to ~ 120 - 200°F. Work carried out at Aerospace³⁶ indicated increases in free radical yields with increasing temperatures during irradiation over this range and slightly higher (~ 250°F). The latter results, obtained for TFE samples irradiated in air, were attributed to increased mobility in the polymer, permitting free radical fragments to diffuse away from one another before recombining. This suggests that mechanical properties would degrade more at higher radiation temperatures over this range, a suggestion supported by experimental results.³⁹

Above these temperatures radicals decay,^{16, 41, 55-57} presumably due to recombination of fragments formed during chain scission, as well as to cross linking. This would tend to lead to more or less degradation of mechanical properties than that observed at lower, above-ambient temperatures, the actual effects depending upon the relative importance of geninate recombination and cross-linking. For FEP, the corssover point occurs at ~ 175°F.^{54, 58} Of course, ultimately, at sufficiently high temperatures, the combination of thermal and radiation effects,^{49, 59-65} or heat alone^{66, 67} can lead to severe polymer degradation.

4. Other Degradation Studies

In addition to polymer damage induced by exposure to various elements of simulated space radiation environments, several additional modes of damage have been investigated. These include damage induced by exposure of the fluorocarbon polymers to glow discharges^{27, 68} and lasers.⁶⁹

B. Charging

There is ample evidence in the literature for charge generation and stabilization in both FEP and TFE polymers on exposure to ionizing radiation. Much of this interest has been stimulated because of the potential use of teflon electrets in condenser microphones, electrostatic recorders and other commercial devices, 2, 70-78 Additional evidence for charge generation and trapping comes from thermoluminescence, 79-81 photo- 82-86 and radiation- 87 induced conductivity, and ESR 19, 57, 77 studies. References cited here include work carried out on FEP and/or TFE in vacuum and/or air.

Charge stabilization occurs from liquid helium⁸⁰ and liquid nitrogen 19, 78, 79, 81 temperatures up to $>350^{\circ}\text{F}$. 71-76 Examination of thermoluminescence⁷⁹⁻⁸¹ data indicates considerable structure in the luminescence or conduction versus temperature curves, suggesting multiple trapping sites. Although charges produced at low temperatures decay rapidly on warming to room temperature, 19, 78-81 those produced at ambient temperatures are stable for periods of weeks to years at those temperatures. 71, 72, 75, 89, 90 Trap depths range from ~ 0.1 to $>2\text{eV}$. 57, 70, 74, 78, 80, 81 The presence of gases, even inert gases, during radiation was shown to be important. 79 Only one paper comments on sample coloration and bleaching;⁸⁰ however, such effects would be expected on the basis of the trap depths.

The identification of trapping sites in these polymers is not well defined. However, it has been suggested that the shallower traps ($<1\text{eV}$) occur in the crystalline, amorphous, and interfacial regions of the polymers, 77, 79, 80, 91 while the deeper traps ($>1\text{eV}$) may be associated with electron attachment to $-\text{CF}_2\cdot$ and $-\text{CF}_2\dot{\text{C}}\text{FCF}_2-$ radicals as well as unsaturated structures. 19, 78, 81

C. Structural Effects

Virtually all of the work carried out on structural studies of these polymers has been done on TFE materials. A wide variety of techniques have been applied to such studies of unirradiated TFE including infrared⁹²⁻⁹⁶ and Raman⁹⁷⁻¹⁰² spectroscopy, x-ray diffraction,¹⁰³⁻¹⁰⁵ nuclear magnetic resonance,¹⁰⁶⁻¹¹² electron microscopy,^{113,114} as well as measurements of specific volume^{3,115,116} and mechanical properties.¹¹⁷⁻¹²² Theoretical calculations have also been carried out.^{123,124} Only four such studies^{54,125-127} have been found on FEP, one of which deals with surface characterization of etched polymers.¹²⁷ Consequently, radiation induced changes in TFE materials will be discussed, followed by comments on FEP polymers. Additional studies on properties of unirradiated fluorocarbon polymers can be found in Refs. 128-144.

The TFE polymers contain varying degrees of amorphous and crystalline regions, depending upon their manner of preparation, that exhibit phase transitions associated with different molecular motions at \sim -143, 66, 86, 260, and 620°F (Refs. 118, 145, 146, and references therein). Upon irradiation, chemical changes produce changes in the amorphous and crystalline structures of the polymers that directly affect macroscopic properties. For example, a number of studies have demonstrated increases in crystallinity upon exposure to ionizing radiation (doses up to 100 Mrads) in vacuum, as inferred by results from experiments utilizing a number of different analytical techniques.¹⁴⁷⁻¹⁵⁴ (The latter results are for samples irradiated in vacuum and measured in air; increases were also observed for samples irradiated and measured in air.^{3,146,154,155-161}) These increases are generally accompanied by a lowering of the transition temperatures noted above.^{3,146,148,152,160,161} Formation of -CF₃ groups has also been observed in irradiated TFE.^{151,152} At higher doses (<300 Mrads), decreases in apparent crystallinity were observed, observed,^{3,148,157,160} as well as embrittlement and cracking.^{3,147,149,151,160}

Additionally, post-irradiation heat treatments led to further increases in crystallinity. ^{3, 148, 151, 155, 157, 159, 160}

These various observations have been interpreted in terms of radiation induced changes on a molecular scale. Increased crystallinity at low doses is generally attributed to chain scission in the amorphous regions of the polymer. ^{146, 149, 153} An additional piece of evidence supporting this conclusion is that low crystallinity TFE is more affected by irradiation than highly crystalline materials. ¹⁴⁹ Because of cage effects and the more rigid structure in the crystalline regions, chain scission in these regions is thought to be followed by geminate recombination, possibly with some disordering. ^{144, 148, 157} Chain scission in the amorphous regions is thought to permit disentanglement and reordering of the polymer chains in those regions into a more ordered, preferred structure. ^{146, 148, 153, 155-157, 160} These events would be accompanied by some reduction in molecular weight ^{148, 149, 155, 160} and, in view of the ESR results described above, some recombination of chain ends. Cross-linking could also occur (e.g., reactions of $-\text{CF}_2\dot{\text{C}}\text{FCF}_2-$ with similar free radicals or with $-\text{CF}_2\bullet$), as has been suggested. ^{146, 149, 156} Post-irradiation heating could facilitate chain rearrangements and/or cross-linking because of increased molecular motions at the higher temperatures, promoting further increases in crystallinity as have been noted. ¹⁵³

At higher doses, percentage changes in the amorphous regions become smaller and disordering in the crystalline regions, because of radiation induced defects and gas occlusion begin to dominate, leading to reductions in crystallinity. ^{146, 148, 157} Integration of these effects and those noted in the preceeding paragraph contributes to a more rigid structure and embrittlement of the polymer at high doses. ^{149, 155} Formation of $-\text{CF}_3$ groups can also contribute. ¹⁵² At lower doses, gases generated by polymer irradiation may migrate to incipient or radiation induced voids in the material. However, chain scission is still rather minimal, and the polymer can accommodate the occluded gases. With higher doses, the internal pressures build up, possibly as high as 300-400 atm³. This, coupled with weakening

of the polymer structure through chain scission processes, can lead to cracking as the occluded gases force their way to the surface. 3, 155, 157, 160

The properties of unirradiated FEP are somewhat similar to those of TFE in that various types of phase transitions are observed, but at different temperatures. 54, 125 One study indicated that nuclear magnetic resonance absorption versus temperature curves, associated with a low temperature ($\sim -40^{\circ}\text{F}$) transition were unaffected by irradiation of FEP. 126 Little is known about the overall effects of radiation on crystallinity, although experimental evidence suggests chain scission, such as occurs in TFE, also occurs in FEP, 16, 17, 54 and might be expected to have similar effects. Cross-linking appears to be quite important in FEP, 20, 39 especially at temperatures above 180°F , 54 and would be expected to lead to more rigid structures and embrittlement. One study suggested cross-linking in the irradiated polymer could be increased by heating or mechanical stress. 54 Gas evolution in irradiated FEP, 20, 49 especially at high doses, might be expected to lead to cracking as is observed in TFE. 3, 155, 157, 160

D. Mechanical Effects

The effects of mechanical stress on fluorocarbon polymers is considered here. These effects will be important in the mechanical testing of such materials exposed to simulated space environments. As in the case of structural studies, virtually all of this work has been carried out on TFE polymers. However, many of the considerations presented here would also apply to FEP materials.

A number of studies on TFE, 162, 163 and other, non-fluorocarbon polymers, 164-170 have demonstrated the production of free radicals upon mechanical stress of these materials. For TFE, 162, 163 unlike the case of irradiation, free radicals resulting from chain scission ($-\text{CF}_2\cdot$) are the dominant species formed upon mechanical fracture of TFE. These scissions are thought to occur at high stress points in tie molecules, i. e., polymer chain molecules traversing the amorphous regions of the polymer and

anchored at both ends in two different crystalline regions. 163, 171-174
Occluded gases might be important with respect to increasing stresses in these regions.

As has been suggested in the case of irradiated polymers subjected to ionizing radiation, ⁵⁴ cross-linking due to reactions of free radicals formed by mechanical fracture might also be expected. These, of course, could also interact with free radicals produced by irradiation of the polymers. Changes in free radical concentrations and crystallinity with irradiation and thermal cycling (Sections II. A. and II. C.) would also be expected to influence results from mechanical tests. So also would the effects of the test temperature, due to free radical changes as well as phase changes in the polymer.

E. Dose Rate Effects

Only a limited number of studies have investigated dose rate effects in fluorocarbon polymers. Electret related studies have indicated that conductivity during pulsed radiation⁸³ and thermally stimulated post-irradiation currents⁷⁶ are independent of dose rate, although conductivity measured during continuous irradiation may exhibit a slight dose rate dependence.¹⁷⁵ However, these studies were carried out in air, generally at low dose rates (<1 Mrad/hr). Additional studies carried out on TFE in air exhibited distinct dose rate effects on dielectric properties¹⁷⁶ and free radical concentrations,¹⁷⁷ which are probably due to the extreme radiation sensitivity of peroxide radicals formed by reaction of $-\text{CF}_2\dot{\text{C}}\text{FCF}_2-$ with oxygen.^{42, 43}

The most relevant studies of interest here concern increased degradation and structural changes observed as a function of dose rate. Degradation of TFE in a helium atmosphere, as measured by evolution of gaseous products, was found to increase as the square root of the dose rate (0.1-7.6 Mrads/hr), in experiments conducted at 625-840°F.⁴⁹

Analysis of results from nuclear magnetic resonance experiments conducted on TFE at dose rates of 0.1 - 1.4 Mrad/hr and temperatures of -100 to 620°F were interpreted in terms of dose rate/temperature effects on polymer structures.¹⁵³ Thus, for irradiations carried out at high temperature with subsequent cooling to room temperature after irradiation, it was suggested that chain scission leading to crystallination (see Section II. C.) could occur at low dose rates where molecules had ample time to reorient during irradiation before cooling to room temperature. For samples irradiated at higher dose rates, at the same temperature and to the same total dose, the reorientation and crystallization would not be as extensive. Conceivably, in the latter case, free radicals that did not reorient could undergo germinate recombination, resulting in a lessor net amount of chain scission.

III. DISCUSSION OF SPACE SIMULATION TEST RESULTS

In a recent series of tests, ^{4,5} the properties of metalized FEP films were examined after exposure to simulated space radiation environments. Applicable conditions in these tests are summarized in Table I. Test A (Table I) was a short-term high exposure level, screening type of exposure, while tests B, C and D were long term, low exposure level experiments. Test results for changes in tensile strength and elongation are summarized in Table II. (For changes in optical properties for these materials, as well as test results for other materials, see Refs. 4, 5.)

Comparison of the results for tests A and B (Table II) indicates more substantiated degradation of mechanical properties in the long-term, low exposure level tests. Major differences between these two tests, aside from the greater acceleration rate for test A, were the greater integrated near UV flux and higher sample temperatures in test B relative to test A. The higher temperature in test B could lead to slightly higher free radical yields (Section II.A.3.). However, subsequent geminate recombination and especially crosslinking would also be expected to be greater in test B, both due to the higher sample temperatures and the longer period of time samples were maintained at those temperatures (Section II.A.3.). Although geminate recombination would lead to restoration of mechanical properties, crosslinking would yield a more rigid structure with attendant changes in mechanical properties, e.g., decreases in ultimate elongation. As noted earlier (Sections II.A.1 and II.A.3), crosslinking in FEP becomes particularly important at temperatures $\geq 175^{\circ}\text{F}$. Additionally the higher temperature and longer duration of test B suggest relaxation phenomena such as discussed in Sections II.D. and II.E. could also be more important for these samples relative to those in test A.

Table I. Simulated Space Radiation Exposures of Metalized FEP Films^a: Test Conditions

Test	A	B	C	D
Reference	4	4	4	5
Equivalent Months of Electron Irradiation in Synchronous Orbit ^b	60	39	39	84
Test Duration	3 days	6.4 mos	6.4 mos	11 wks
Average Electron Acceleration Rate ^c	600	6	6	35
Near UV Acceleration Rate ^d	nominal	1.5	2.5	2
Sample Temperatures During Test (°F)	150	200	100	≤15 ^e
Mechanical Properties Tested ^f	in situ	in situ	ex situ	in situ

^a 2-mil FEP Type A Teflon plastic films coated with vacuum-deposited silver overcoated with Inconel.

^b Simulated with 80 + 120 kev electrons (A, B, C) or 125 + 350 kev electrons (D).

^c Electron exposures were periodic during the duration of the tests so that instantaneous acceleration rates were higher, especially for tests B and C and, to a lesser extent for test D.

^d 0.18 - 0.40 μ m. Approximately the same UV flux was used in test A as in tests B, C and D. However, integrated near UV exposure was considerably less.

^e Samples maintained at 15°F except that one day a week they were temperature cycled to -60 to -100 ± 10°F with a concomittant reduction in the near UV acceleration rate to 0.1X.

^f In situ samples tested in the test vacuum environment. Ex situ samples tested in air after vacuum irradiation.

Table II. Changes in Mechanical Properties of Metalized FEP Films
Subjected to Simulated Space Radiation Environments

Test	Equivalent Months in Synchronous Orbit ^a											
	<u>0</u>	<u>6</u>	<u>9</u>	<u>12</u>	<u>17</u>	<u>18</u>	<u>24</u>	<u>30</u>	<u>36</u>	<u>39</u>	<u>60</u>	<u>84</u>
Ultimate Tensile Strength (PSI)	A	4020									1850	
	B	4020	2500			2060	1750	X		X		
	C	3470	2130	1920	(1000) ^b				X			
	D	NA					X		X			
Ultimate Elongation (%)	A	457									30	
	B	457	45			2	1.7			X		
	C	473	5.3	3.6	(1.5) ^b							7.7 ^c
	D	NA					X		X			

^a X = samples failed before reaching indicated total exposure; NA = not available at this writing, but presumably comparable to other non-exposure values.

^b Here only, one sample. Two other samples failed at this total exposure. Other data for test C are averages of three samples.

^c Near UV exposure only, this sample only.

Thus the picture that emerges from consideration of thermal effects is that there was probably a greater degree of net chain scission in test A samples, with increased crosslinking and relaxation (crystallization) in test B samples. Test results (Table II) indicate the latter effects were more important in producing degradation of mechanical properties.

Of course, the test B samples experienced a greater integrated exposure to near UV. As noted earlier (Sections II.A.1 and II.A.3), near UV exposures can lead to chain scission at temperatures $< \sim 175^{\circ}\text{F}$ and crosslinking at temperatures $> \sim 175^{\circ}\text{F}$. (Evidence for direct UV photolysis of the fluorocarbon polymers can be seen in the test D results in Table II, as well as Refs. 50-54). These considerations lead to similar conclusions to those discussed in the preceding paragraph for strictly thermal effects.

Several attempts were made to determine the relative contribution of thermal and near UV effects to degradation of mechanical properties noted in Table II. A simple three step model was derived involving radiation induced free radical production, free radical recombination, and a thermally induced free radical transformation (chemical or physical such as relaxation of the free radical polymer molecule to a crystalline configuration). Crude estimates of rates for these various processes could be obtained from the literature cited in Section II. Results from the model calculations were qualitatively consistent with the data in Table II. Unfortunately, within the experimental uncertainties for the rates of the various processes, results from calculations wherein the third step was UV induced rather than thermally induced, were also consistent with the data. The literature data do indicate that thermal effects would be significant in contributing to the differences in results from test A and B. The role of UV exposures can best be described as small (e.g., $\sim 10\%$) to comparable to the thermal effects.

The lower temperature in test C relative to test B (100°F versus 200°F) would be expected to lead to sharply reduced geminate recombination and crosslinking in test C samples as well as greater net chain scission (Sections II.A.1 and II.A.3). Relaxation and reorientation of polymer

segments should be less. It would appear then that chain scission is the dominant process contributing to the results from test C. Similar to the case of comparison of results from tests A and B, the higher UV flux used in test C, which would also produce greater chain scission, does not permit an accurate determination of the relative contributions of thermal and UV effects to the results in tests B and C.

The quantitative information from test D is very limited, but suggestive of even greater degradation of properties than was experienced in test C. Since both the UV flux and integrated exposure were less in test D, the differences between the test C and D results appear to be due primarily to the lower temperatures in test D, leading to the effects described in the preceding paragraph. (Note however, that the UV exposures are likely to contribute to degradation in both tests C and D; only the differences in the results appear to be explainable solely on the basis of thermal effects.)

Although the results in Table II appear to be explainable in terms of temperature and UV effects, dose rate effects cannot be entirely ruled out (see also Section II. E). In view of the available literature, it appears that, except at very high dose rates (much greater than those used in these tests), these will be small compared to thermal and UV effects.

IV. CONCLUSIONS AND RECOMMENDATIONS

A. Application of Existing Data to Spacecraft Problems

The inapplicability of data for fluorocarbon polymers obtained from experiments conducted in the presence of oxygen (air) to spacecraft problems has been discussed by a number of authors (Section II and references therein). One of the major conclusions resulting from a review of the literature is that thermal effects can play a very important role in material degradation and must be considered in any application of existing data. Thus, for example, above $\sim 175^{\circ}\text{F}$, radical recombination and crosslinking (Sections II. A. 1 and II. A. 3), as well as mechanical relaxation (Sections II. C and II. E) can be important in irradiated FEP polymers, particularly in long term exposure tests at elevated temperatures. Application of data from such exposures to a spacecraft environment where temperatures might not exceed, say 50°F , could result in misleading conclusions regarding the stability of these materials in the actual environment. The same would be true for TFE materials (Sections II. A. 1 and II. A. 3). Extrapolation of higher temperature data to lower temperatures is not straightforward, since the various processes that can occur are likely to have substantially different temperature coefficients.

Data reviewed earlier (Section II, especially Refs. 39 and 54), as well as that in Table II indicate that for FEP materials high temperature data, depending upon the nature of the experiment, may serve only as a lower limit to material damage that would be experienced at lower temperatures, while the opposite may well be true for TFE materials (e.g., tensile strength and elongation decreased significantly for TFE irradiated in vacuum at elevated temperatures³⁹).

Similarly, application of ionizing radiation data only, to the estimation of space radiation effects is likely to result in a lower limit to damage that would actually be experienced. Ultraviolet radiation can degrade FEP polymers by itself (Sections II. A. 1 and II. A. 3, especially Refs. 50-54, and Table II, test D). The latter may also act synergistically with ionizing radiation, although evidence for this is not clearly established. Such effects might be especially important at lower temperatures where there is greater stabilization of trapped charges (Section II. B).

Overall, it is difficult to quantify these statements due to variabilities in temperature, exposure rates and other parameters that could affect net degradation in the experiments that have been carried out to date. However, the data in Table II and references in Section II suggest that deterioration of FEP films in an actual low temperature ($\sim 20^{\circ}\text{F}$) space environment will occur at a rate approximately 3-10 times that observed for accelerated (e.g., 500X) ionizing radiation (only) exposure rates at elevated temperatures (e.g., $\sim 150\text{-}250^{\circ}\text{F}$). Ultraviolet effects on top of deterioration from ionizing radiation exposures only could increase deterioration rates by perhaps 10% to a factor of two. These values are subject to considerable uncertainty. They are offered only as very crude numbers for estimating effects, and not as a substitute for actual relevant laboratory results upon which actual decisions concerning these materials should be made.

B. Future Space Environmental Exposure Tests

From the discussion in Sections II and III, it is apparent that two of the crucial variables that must be carefully controlled in any future tests are the radiation dose rate and temperature. As discussed in Section III, the greater deterioration of mechanical properties observed in the long term, low level radiation exposure of FEP film relative to short term high level exposures is probably due to the extended time samples were maintained at elevated temperatures, rather than true dose rate effects. Quite different results would probably be obtained for the short term, high exposure level samples if they were maintained at elevated temperatures for extended periods of time after irradiation.

Temperatures should simulate as nearly as possible those anticipated in space. Moreover, samples should not be exposed to excessive temperatures after radiation since thermal effects can be induced even after termination of the radiation exposure (Section II). Additionally, the lack of definitive data on the possibility of synergistic effects between ionizing and UV radiation suggests that both should be used in any future simulation studies.

Where temperature cycling is employed, exposures terminated on the low temperature end of the cycle will probably give the worst case results, since some annealing of low temperature damage could occur at the high temperature end of the cycle. One final comment relates to mechanical testing of irradiated samples. As noted in Section II D., mechanical stress itself can induce damage in fluorocarbon polymers. Free radicals produced by mechanical stress could interact with those produced by irradiation. Alternatively mechanical stress could cause slippage of polymer substructures, altering the spatial distribution of radiation induced free radicals, that could lead to more or less recombination. For these reasons it is recommended that mechanical tests be performed on a series of samples irradiated to different exposure levels, rather than a single set of samples irradiated to different levels and tested after each exposure increment.

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Teflon literature categories are given in right-hand column of list.

- A - STUDIES WITH FEP POLYMERS
- B - STRUCTURE AND/OR MECHANICAL PROPERTIES - IRRADIATED TEFLON
- C - STRUCTURE AND/OR MECHANICAL PROPERTIES - UNIRRADIATED TEFLON
- D - ELECTRICAL PROPERTIES OF TEFLON - GENERAL
- E - ELECTRICAL PROPERTIES - PHOTO- AND THERMO-CONDUCTIVITY IN TEFLON
- F - ELECTRICAL PROPERTIES - ELECTRETS IN TEFLON
- G - OPTICAL PROPERTIES AND LUMINESCENCE OF TEFLON
- H - GASEOUS EVOLUTIONS IN AND INTERACTIONS WITH TEFLON
- I - FREE RADICAL PRODUCTION AND/OR STUDIES OF TEFLON
- J - LIGHT INDUCED FREE RADICAL TRANSFORMATIONS IN TEFLON
- K - OTHER STUDIES WITH TEFLON OR RELEVANT STUDIES WITH OTHER POLYMERS

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I

THE FOLLOWING REFERENCES CONTAIN ADDITIONAL INFORMATION ON PROPERTIES OF
 UNIRRADIATED AND IRRADIATED TEFLON AND RELATED MATERIALS, AS WELL AS
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